CAPSULATION OF MOLDINGS MADE FROM SILICON CERAMIC MATERIAL

Axel Rossmann, Klaus Schweitzer, Werner Hueter

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7. Author(s)
Axel Rossmann, Klaus Schweitzer, Werner Huether

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Leo Kanner Associates
P.O. Box 5187
Redwood City, CA 94063

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16. Abstract
Ceramic articles are potted for hot isostatic pressing by porous glass and/or ceramic coating which is sintered to a pressure-tight coating in vacuo. Thus, a powered SiO_2 glass mixture with saturated alcohol sterin is sprayed on a SI_3N_4 ceramic, dried, introduced into the press which is evacuated to less than 0.013 mbar. and heated to approximately 1200° to drive off the organic binder and leave a powdered glass coating on the ceramic. The coating is sintered by heating to approximately 1200° for 0.5-2 hours and forms a tight gas-impermeable layer. The press is heated to approximately 1700° at 1000-300 bar and isostatic pressing is performed in the conventional manner.

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Axel Rossmann, Klaus Scheitzer, Werner Huether

Patient Claims

1. Method for capsulation of a ceramic mold,* especially silicon ceramic by means of a glass layer surrounding the mold for the hot isostatic pressing (HIP), characterized by the fact that a porous permeable layer of glass and/or ceramic is applied on the mold and sintered under vacuum into a pressure-tight capsule enclosing the mold.

2. Method for capsulation of a reaction sintered silicon ceramic mold, especially of silicon nitride ($Si_3 N_4$) which still contains excess silicon, characterized by the fact that a permeable porous layer of glass and/or ceramic is applied on the mold, the mold is then evacuated and filled with nitrogen (N_2) and then the layer is sintered into a pressure tight capsule.

3. Method according to claims 1 to 2, characterized by the fact that powders consisting of an SiO_2 glass, as SiO_2-B_2O_3 of glasses of these types with additives of MgO or Al_2O_3 are used for the porous layer.

4. Method according to claims 1 or 2 or 3, characterized by the fact that glass powder containing lithium is used for the porous layer.

5. Method according to one of the previous claims, characterized by the fact that a mixture of glass powder and an organic binder is applied on the mold and the porous layer is produced by drying of the mold coated in this manner.

6. Method according to one of the claims 1 to 4, characterized by the fact that porous layer is supplied by thermal frame on the mold.

7. Method according to claim 5, characterized by the fact that the organic binders used are saturated alcoholic stearin solutions or aqueous

*Editor's Note: "mold" should be "molding" throughout the text.

gelatin paste.

8. Method according to claim 1 to 2 and one or several of the claims 3 to 7, characterized by the fact that the sintering of the porous layer is conducted in a temperature range of 700 to 1400° C.

9. Method according to claims 1 to 2 and one or several of the previous claims, characterized by the fact that the porous layer is tightly sintered for half to 2 hours.

10. Method according to claim 1 or claim 2 and one or several of the other claims, characterized by the fact that the tight sintering is conducted in a hot isostatic press and the hot isostatic pressing (HIP) is conducted immediately after sintering.

The invention concerns a process for capsulation of a ceramic mold, especially silicon ceramic, by means of a glass layer surrounding the mold for hot isostatic pressing. In the hot isostatic pressing (HIP) porous silicon ceramic molds are compressed under simultaneous application of high pressure acting all-round and high temperature. Through the hot isostatic pressing both mechanical strengths at high temperature and resistance to oxidation can be improved by reducing the porosity of the molds. The pressures applied in hot isostatic pressing (HIP) are around the order of magnitude of 3,000 bars, the temperature at 1750° C. Gas is normally used as pressure transmitting medium. To prevent this gas from penetrating into the porosity of the mold, the latter must be capsulated pressure tight. Up to now this capsulation was conducted in such a way that the ceramic mold is enclosed in an evacuated glass container, while the glass container is converted to a viscous form in the hot isostatic pressing and is applied as a glass layer on the ceramic mold.

In this known process for capsulation of the ceramic mold, there is danger

that in case of too low viscosity of the glass contained melting in the hot isostatic pressing, the glass layer may penetrate into the porosity of the mold, while under certain circumstances a chemical reaction of the glass with the silicon cermatic of the mold also takes place. Thus the strength of the mold in the superfacial region can also be reduced inadmissibly. Moreover through too intense penetration of the thin liquid glass into the porosity of the mold such an intimate binding takes place between the glass envelop and the mold that when removing the glass envelop after the hot isostatic pressing, damages to the ceramic mold, especially those with complicated thin-walled shapes are not avoidable. On the other hand there is danger that for too high viscosity of the glass container melting in hot isostatic pressing and the glass layer applied therefore on the ceramic mold, deformations of the latter would occur which is absolutely undesirable.

The problem of this invention is therefore, to develop a method for capsulation of a ceramic mold, with which a pressure tight capsulation of the mold is achieveable, without obtaining in this connection a too intimate binding of the capsulation material with the material of the mold, but at the same time maintaining exactly the original geometrical shape of the ceramic mold as far as it is at all possible.

To solve this problem according to the invention it is proposed that a permeable porous layer of glass and/or ceramic is applied on the mold and is sintered under vacuum into a pressure tight capsule enclosing the mold.

According to the process based on the invention therefore the materials for the capsulation layer is applied before the evacuation of the molds, Because of the porosity of the applied layer the evacuation can take place afterwards, which represents a considerable simplification under the process technology aspect,

Since according to the process phase of the invention the pressure tight capsulation is completed already before beginning the hot isostatic pressing, the capsulation material can be chosen in such a way that its viscosity remains so high at the extremely high temperatures and pressure prevailing in the hot isostatic pressing, that a penetration into the pores of the ceramic mold need not be feared. On the other hand the high viscosity of the capsulation material in hot isostatic pressing does not constitute any danger or an undesired deformation of the ceramic molds, since the capsule material is shaped before beginning the hot isostatic pressing and the ceramic mold and is compressed only together with it. Another important advantage of the process based onthe invention is that the capsulation can take place also in the HIP press.

Another method of solving the problem which also has the above listed advantages and which is suitable for the capsulation of a mold made of reaction sintered silicon ceramic, especially silicon nitride with the excess of silicon is characterized according to the invention by the fact that the permeable porous layer of glass and/or ceramic is applied on the mold, the mold is then evacuated and filled with nitrogen (N_2), and thereafter the layer is sintered into a pressure tight capsule. In this process the sintering of the porous layer applied takes place accordingly under normal pressure, while the nitrogen gas enclosed in the mold reacts with the silicon and is converted into silicon nitride. The sintering process therefore need not be carried out in a vacuum chamber.

Powders of pure SiO_2 glass, SiO_2-B_2O_3 glasses or glasses of these types with additions of MgO or Al_2O_3 should be used preferably for the porous layer. The B_2O_3 components of such glasses may be between 2 and 15%.

In the further implementation of the invention it is proposed that glass powder containing Li be used for the porous layer. When using glasses containing Li during the increase of temperature in the course of the sintering process an increase of the melting point takes place for example by recrystallization so that a particularly high viscosity of the capsule layer is achieved and therefore the undesirable penetration of the layer into the porous body can be avoided with certainty in the HIP process.

Moreover in a preferred variant of the process the porous layer is produced in a process by which a mixture of glass powder and an organic binder is applied on the mold and the porous layer is produced by drying of the mold coated by this means. The drying takes place at temperatures around 300^0 C. The organic binders used should preferably be saturated alcholic stearin solutions or aqueous gelatin paste or other known plasticizers in sintering technology.

In another preferred variant of the invention the porous layer should be applied on the mold by thermal spraying.

The sintering of the porous layer should preferably be conducted within a temperature range between 700 and 1400^0 C, so that the temperature lies still sufficiently below the temperature of decomposition of silicon nitride. The porous layer should preferably be sintered tight for half to to 2 hours.

The tight sintering is conducted preferably in a hot isostatic press and the hot isostatic pressing (HIP) should be carried out immediately after sintering. A variant of the process based on the invention is described below as an example: first a mixture of SiO_2 glass powder and a binder consisting of saturated alcoholic stearic acid solution is produced and sprayed on the ceramic mold. The applied layer is dried and the mold is then introduced into a hot isostatic press. The press chamber is evacuated to a pressure

P less than 0.013 mbar and subsequently heated to a temperature T_1 (about 1200°). During the heating the organic binder is expelled from the layer. There remains a powdery glass layer, which surrounds the mold. This layer is sintered tight after reaching the temperature T_1 for a period of half to 2 hours and forms a closed gas-tight skin. After the stand the hot isostatic press is heated to the press temperature T_2 of about 1700° and the pressure in the hot isostatic press is increased to 1000 to 3000 bars. The further course of the hot isostatic pressing takes place in the usual manner.